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Preparation and Characterization of Nitinol Bone Staples for Cranio-Maxillofacial Surgery

Z. Lekston, D. Stróż, and M. Jędrusik-Pawłowska

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The aim of this work was to form NiTi and TiNiCo body temperature activated and superelastic staples for clinical joining of mandible and face bone fractures. The alloys were obtained by VIM technique. Hot and cold processing was applied to obtain wires of required diameters. The martensitic transformation was studied by DSC, XRD, and TEM. The shape memory effects were measured by a bend and free recovery ASTM F2082-06 test. The superelasticity was recorded in the tension stress-strain and by the three-point bending cycles in an instrument equipped with a Hottinger force transducer and LVDT. Excellent superelastic behavior of TiNiCo wires was obtained after cold working and annealing at 400–500 °C. The body temperature activated shape memory staples were applied for fixation of mandibular condyle fractures. In experiments on the skull models, fixation of the facial fractures by using shape memory and superelastic staples were compared. The superelastic staples were used in osteosynthesis of zygomatico-maxillo-orbital fractures.

Keywords maxillofacial surgery, nitinol bone staples, osteosynthesis, shape memory alloys

1. Introduction

NiTi shape memory alloys with a chemical composition consistent with ASTM F2063-05 have been approved as metal biomaterials and are used in production of medical implants and devices (Ref 1-3). Products of NiTi alloys exhibiting phenomena of shape memory and superelasticity are widespread in such applications as orthodontic archwires, staples for osteosynthesis, stents, endodontic and surgical instruments (Ref 4-6). These implants show good mechanical properties, high corrosion resistance, and biocompatibility (Ref 7, 8). It is well known that improvement of shape memory and superelastic properties can be achieved by ternary additions and thermal or thermomechanical treatments (Ref 9-12). The NiTi staples applied to internal osteosynthesis in orthopedics and maxillofacial surgery operate usually as the shape memory staples activated by patient body heat (Ref 13, 14). For fixation of craniofacial bone fractures, the NiTi staples that are superelastic at the room temperature can also be used (Ref 15).

The studied NiTi-based implants were prepared at the Institute of Materials Science at the University of Silesia. They were applied to osteosynthesis of craniofacial bone fractures in

the clinical experiments carried out by Department of Cranio-Maxillofacial Surgery of Medical University of Silesia in Katowice. Staples made of $\text{Ti}_{50}\text{Ni}_{48.7}\text{Co}_{1.3}$ obtained agreement of the Bioethical Commission to be used for fixation of mandible bone fractures and were applied in 1990-1995 in clinical applications (Ref 16). Positive results of experiments carried out first for animals and then in human body applications showed that the NiTiCo implants as small staples may be used, for example, for fixation of mandibular condyle fractures or osteosynthesis of zygomatico-maxillo-orbital fractures.

Recently, it was confirmed that the NiTi alloys with Co additions in the range of 1-2 at.% should be considered for future medical applications (Ref 17).

The aim of these studies was to form NiTi and TiNiCo body temperature activated and superelastic staples for clinical joining of mandible and face bone fractures.

2. Materials and Experiments

In the experiments two types of alloys were used: commercial NiTi Euroflex wires (Ti-50.8 at.% Ni) with diameters of 1 to 1.4 mm and wires made of TiNiCo alloys of our own production by vacuum induction melting. The melted ingots were homogenized at 900 °C for 48 h under vacuum of about 10^{-5} Torr and followed pack hot rolled on the shape mill at 900-800 °C to rods with diameters of about 4 mm. After removing surface oxides, the rods were rotary forged to about 2 mm diameters and subsequently hot or cold drawn in sintered carbides wire drawing dies to wires with various diameters.

Samples of the wires 70 mm long and of different diameters were polished, washed in distilled water, scoured in acetone, and then subjected to proper heat treatment. They were solution treated at 700 °C for 15 min, cooled down in iced water, and then annealed at the temperature range of 300-500 °C for 15, 30, and 60 min. The cold worked wires with total deformation

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of 20-50% were annealed in the range of 300-600 °C in air atmosphere. Short annealing times from 5 to 15 min were used. Colored oxide layers were removed by chemical etching in water solution of HF and HNO₃.

X-ray phase analysis was carried out using Philips X'Pert diffractometer with graphite monochromator on the diffracted beam. Cu K_{α1} radiation was used. Phase transformation courses were studied by the DSC-7 Perkin Elmer calorimeter in the temperature range -100 to +80 °C. Cooling and heating speed was 10°/min. Shape recovery was studied for the wires deformed at low temperature (in martensite state) by bend-free recovery according to ASTM F 2082-06 test (Ref 18). The superelasticity and shape memory properties were measured in the elongation tests using Instron 4469 machine and in cyclic three-point bending tests using self-constructed machine. Compression forces of the staples were measured using digital FG-5000A gage.

3. Results and Discussion

X-ray phase analysis of the NiTi wires annealed at 700 °C for 15 min followed by cooling in iced water showed that despite applying argon atmosphere during annealing the wire surfaces were slightly oxidized. Weak diffraction lines for TiO₂ were observed (Fig. 1a). The oxide layer was totally removed after chemical etching in water solution of HF and HNO₃. The diffraction patterns showed the B2-phase lines only (Fig. 1b).

Superelastic properties of NiTi wires recorded at room temperature by tension tests indicated that there existed long plateau up to 8% deformation (Fig. 2). Superelastic properties of these wires in the three-point bending tests were confirmed (Fig. 3). On all recorded force-deformation plots, the plateau of forces during loading and unloading were observed.

In the as-received wires no transformation effects were observed in the DSC curves, the heat effects appeared for wires annealed in the temperature range 400-600 °C (Fig. 4).

In the specimen annealed at 400 °C one distinguished peak was observed at about 40 °C on cooling and two wide, hardly separated peaks could be seen on heating between 10-40 °C. This proves that the transformation course is B2 ⇌ R ⇌ B19'.

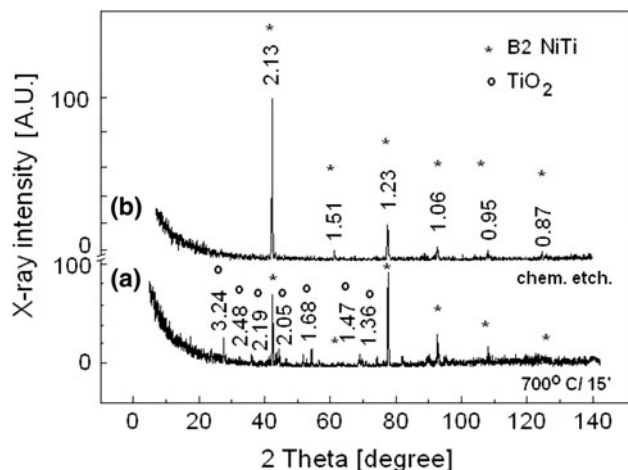


Fig. 1 X-ray diffraction patterns of NiTi wire after solution treatment and after chemical etching

Similarly, two stage transformations were observed for specimens annealed at 500 °C. Increasing the annealing temperature causes that the transformation occurs directly from the B2 to B19' martensite phases. It is worth to mention that for the specimen annealed at 400 °C the characteristic temperatures are higher than for the wires annealed at 500 and 600 °C. This can be explained by interaction of recovery and precipitation of the Ni₄Ti₃ phase processes.

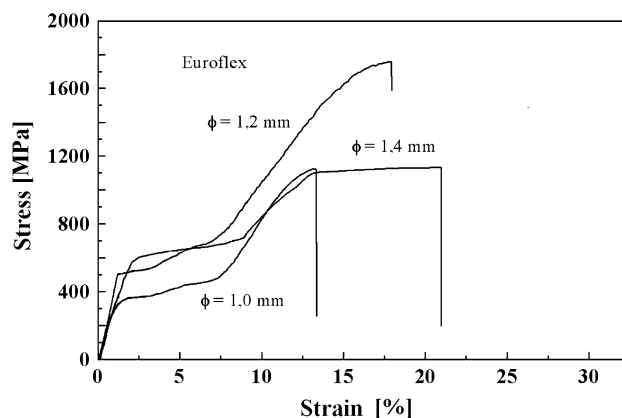


Fig. 2 Stress-strain tests of NiTi as-received wires

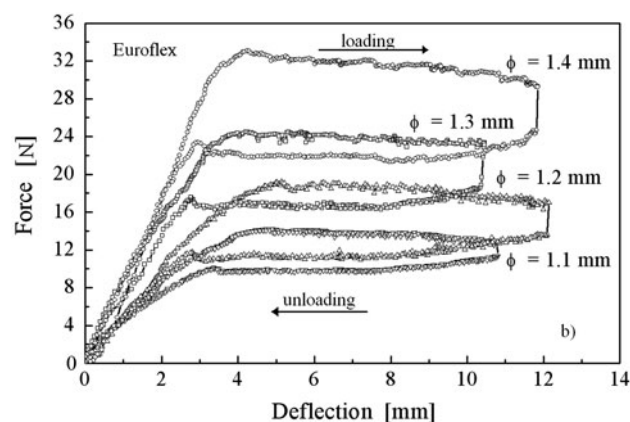


Fig. 3 Load-deflection plots of NiTi wires recorded during a three-point bending deflection

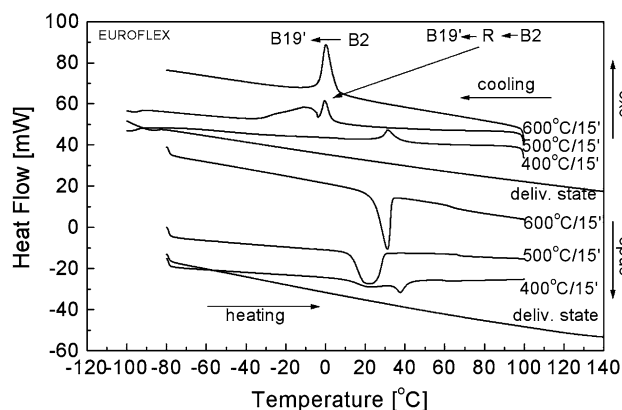


Fig. 4 Phase transformation in NiTi wires after cold drawing and annealing

Shape recovery measurements carried out according to the ASTM F 2082-06 standard showed that the as-received wires recovered at too low temperatures, i.e., $A_f \sim -20^\circ\text{C}$ (Fig. 5a). Implants working at the body temperature require much higher recovery temperature. This was obtained by annealing the samples for 0.5 h at 400–600 °C for which the recovery occurred at about 40 °C (Fig. 5b).

XRD analysis proved that also the NiTiCo alloys were in the B2-phase at room temperature (Ref 19). Cold drawing followed by annealing caused that on the X-ray diffraction patterns splitting of the 110_{B2} reflection appeared. This means that the R-phase occurred in the specimens (Ref 20, 21). This was confirmed by TEM studies (Fig. 6).

This is typical for the alloys deformed and then annealed with specific dislocation structure (Ref 22). The recovery process causes the dislocation cellular structure, i.e., in the specimen two regions can be distinguished: with low dislocation density—inside the cells; and high dislocation density—in the cell boundaries. The transformation occurs in these two regions at different temperatures.

Also the DSC curves for the specimens annealed after cold drawing show multi-stage character of the transformation (Fig. 7). For specimen annealed at 500 °C two distinguished, well-separated peaks of the R-phase and then B19' transformations were observed. Annealing at lower temperature caused also that both transformations occurred each in two stages.

The shape recovery curves of the TiNiCo hot drawn wires, solution treated from 750 °C and additionally aged at 400 °C for 1 h show that recovery occurred at about 50 °C (Fig. 8). This is too high for implants activated by human body

temperature. These wires can be used as implants activated by outer heat source (e.g., warm physiological salt).

Good temperature of shape recovery was obtained for the cold drawn wire of $\text{Ti}_{50}\text{Ni}_{48.7}\text{Co}_{1.3}$ annealed at 400 °C for 15 min (Fig. 9). These wires were used to prepare staples for fixation of broken bones, activated by human body temperature, 37 °C.

In the wires of similar chemical composition subjected to high deformation followed by annealing good superelasticity was obtained (Fig. 10).

These alloys were used to prepare staples that could be easily inserted in the bone holes by mechanical bending their legs (Fig. 11). The staples for fixation of bone fractures were

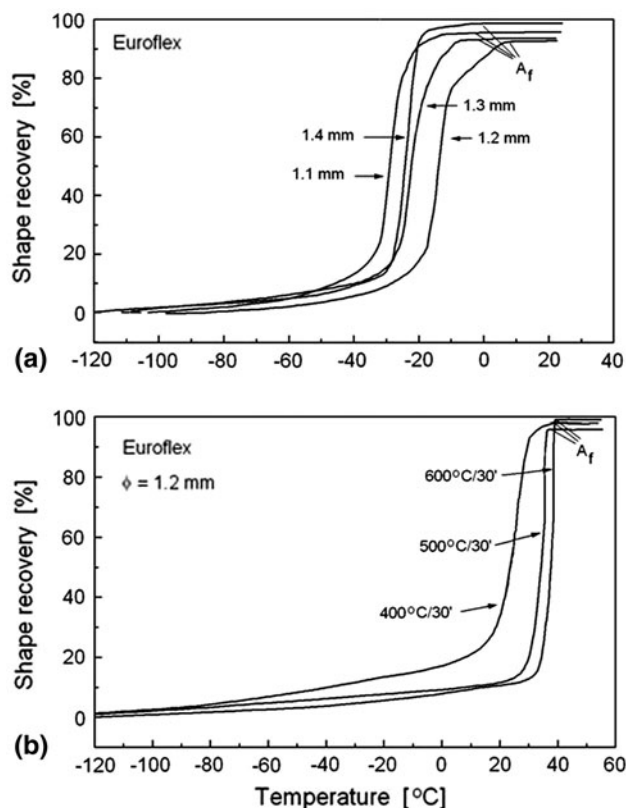


Fig. 5 Shape recovery curves of different diameter wires in delivered state (a) and of 1.2 mm diameter wire after recrystallization annealing (b)

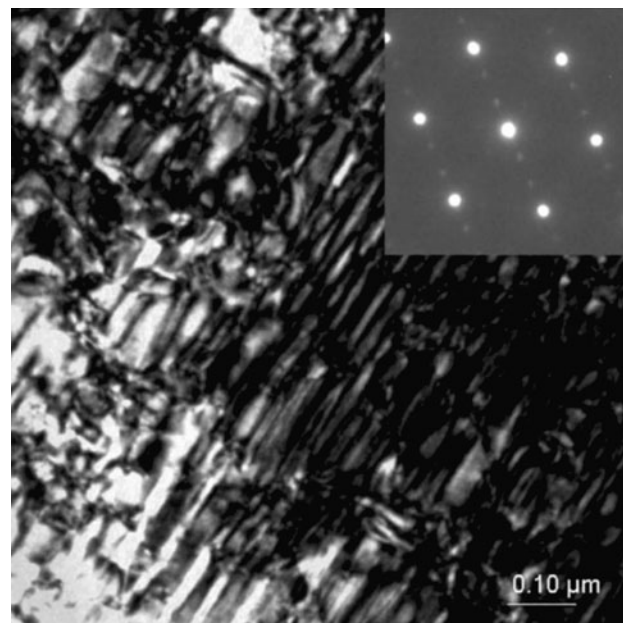


Fig. 6 R-phase structure in the NiTiCo annealed at 400 °C. The diffraction pattern shows additional $1/3 \langle 110 \rangle_{\text{B2}}$ which are typical for the R-phase

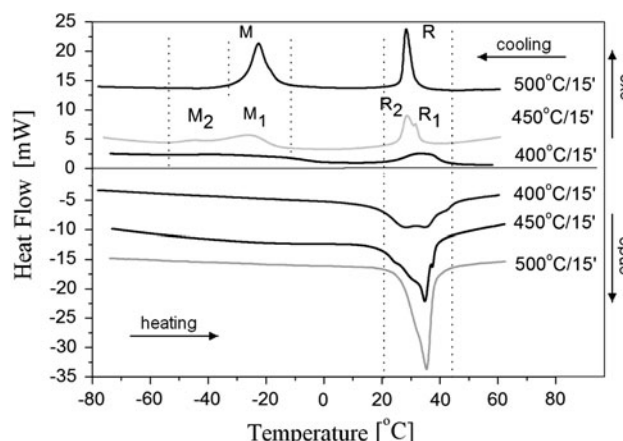


Fig. 7 Phase transitions in TiNiCo wires after cold drawing and annealing

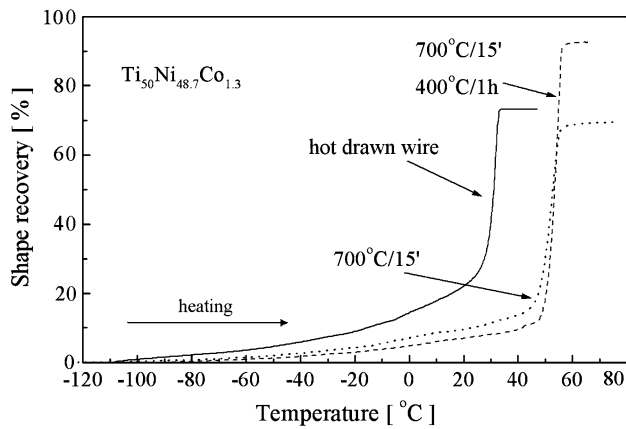


Fig. 8 Phase transitions of TiNiCo hot drawn wire and wires after solution treatment and aging

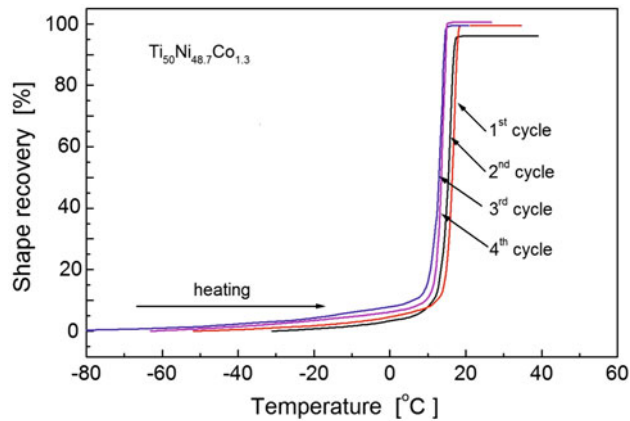


Fig. 9 Shape recovery curve of TiNiCo wire after most favorable cold drawing and annealing

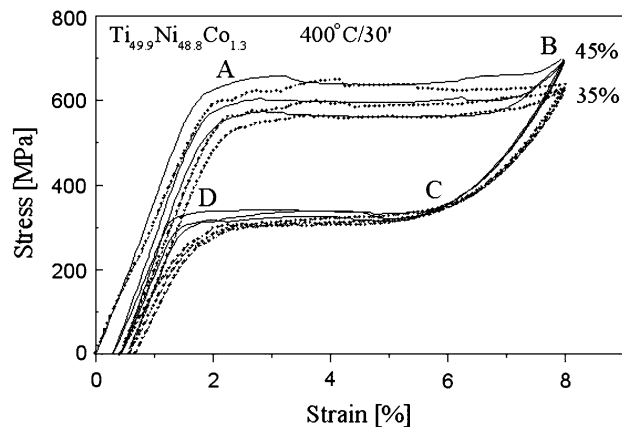


Fig. 10 Stress-strain curves of TiNiCo wires after cold drawing and annealing

formed under the flame of gas burner by bending their ends up using the pliers for forming the wires. From wires with

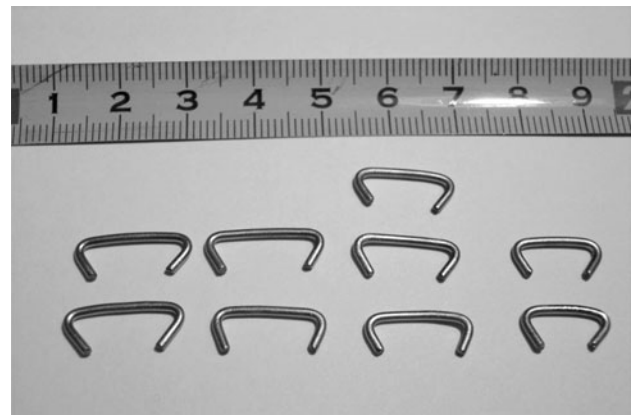


Fig. 11 Superelastic staples for fixation of face bone fractures

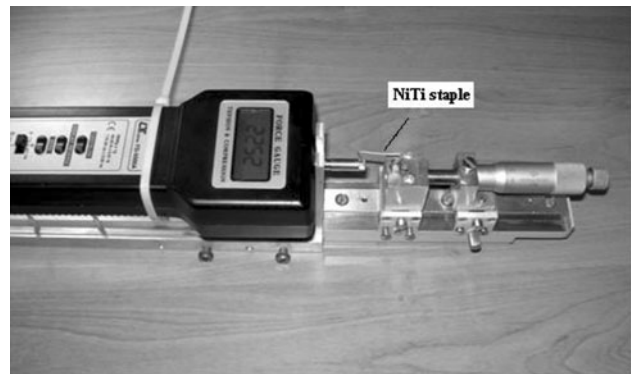


Fig. 12 Digital force gage for measurements of forces at bending the staple legs up to 90°

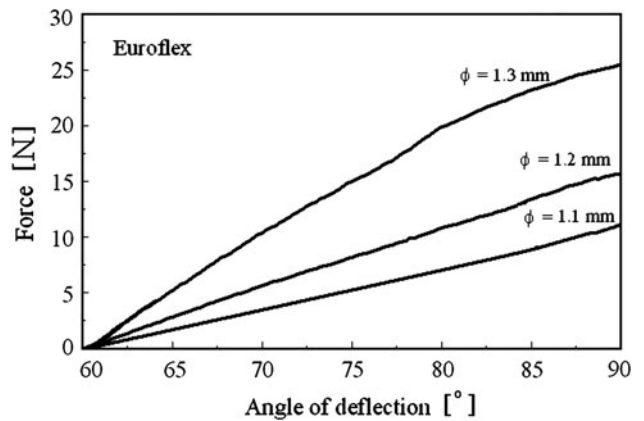


Fig. 13 Changes of forces during deflection of legs of NiTi staples made from wires with various diameters

diameter 1.3, 1.2, and 1.1 mm the staples with the length of the span between 7 and 15 mm were formed.

Measurements of compression force of superelastic staples were recorded by using digital force gage (Fig. 12). The staples were opened at room temperature up to 90° angle between the legs and the span.

The compression forces of NiTi staples versus the deflection angle are shown in Fig. 13. The forces of prepared superelastic NiTi and TiNiCo staples in dependence of length of span, diameters of wires, and angles of legs were comprised in a range from several to about 30 N.

Recently in the Department of Skull and Maxillofacial Surgery of Silesian Medical University in Katowice carries out research on the development in implementation of a new method of subcondylar mandible fracture fixation by means of NiTi shape memory staples.

In the laboratory test it appeared that if the fixation is made by a single staple (Fig. 14a), the broken mandibular condyle may slip (Fig. 14b). It has been shown that better stability of the

fixation of mandibular condyle fracture is secured by two parallel shape memory staples (Fig. 14c). After analysis by using the finite element method, for stabilization of subcondylar fractures, two shape memory staples were used in each case (Ref 23).

The use of NiTi superelastic staples for joining of zygomatic and Le Fort I face bone fractures is demonstrated on the model of the cranium (Fig. 15a, b).

Prepared NiTi shape memory staples with desired dimensions and properties after their laboratory studies were selected and applied for fixation of low subcondylar fractures of mandible of patients with multiple condyle mandible fracture (Ref 24). The superelastic staples were used for joining of zygomatic bone fracture.

An example of joining of multiple condyle fracture by using a body heat-activated NiTi staples and postoperative radiogram which proves an anatomical position of joined bone fragments is shown in Fig. 16.

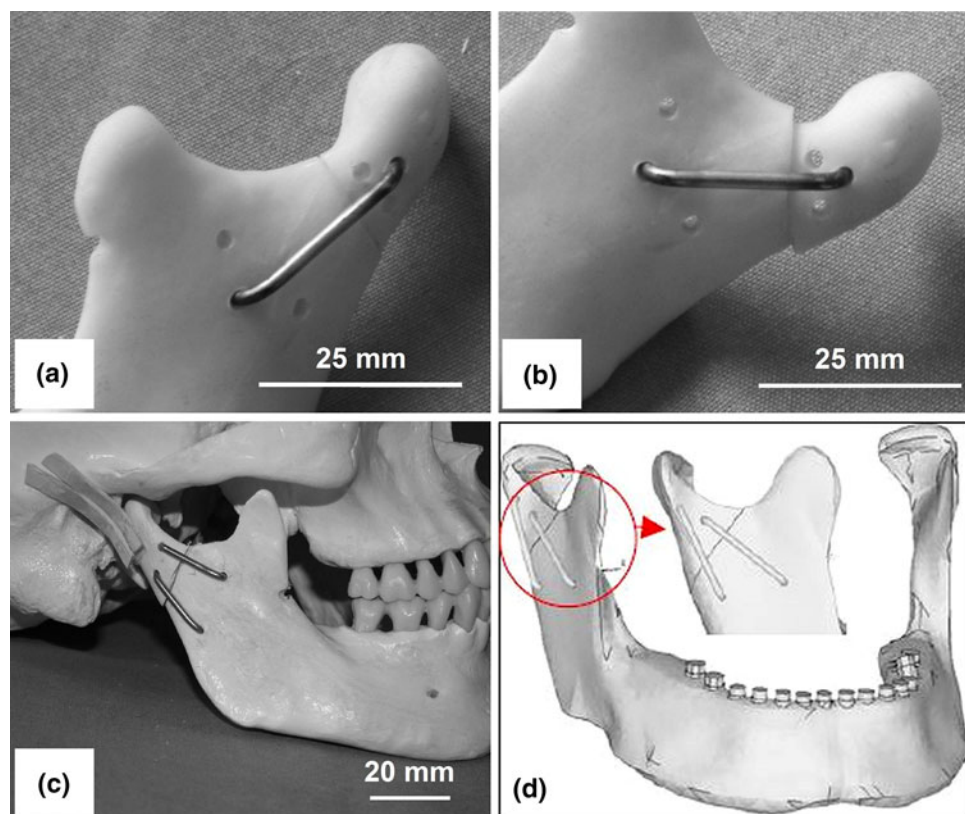


Fig. 14 Experimental fixations of low subcondylar fracture (a-c) and model of mandible for FEM analysis (d)

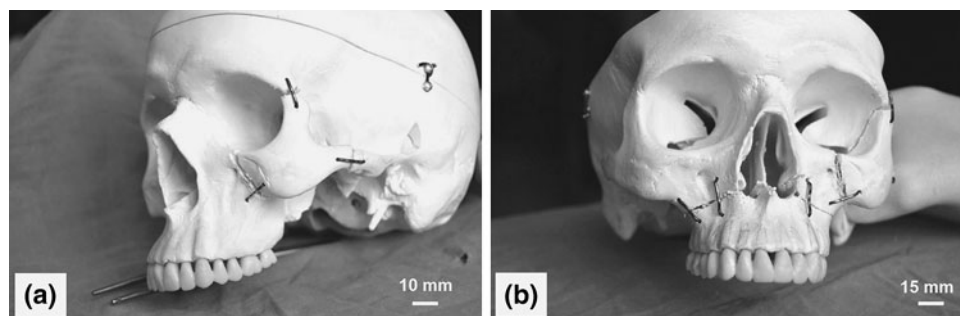


Fig. 15 Experimental fixations of zygomatico-maxillo-orbital (a) and Le Fort I bone fracture (b)

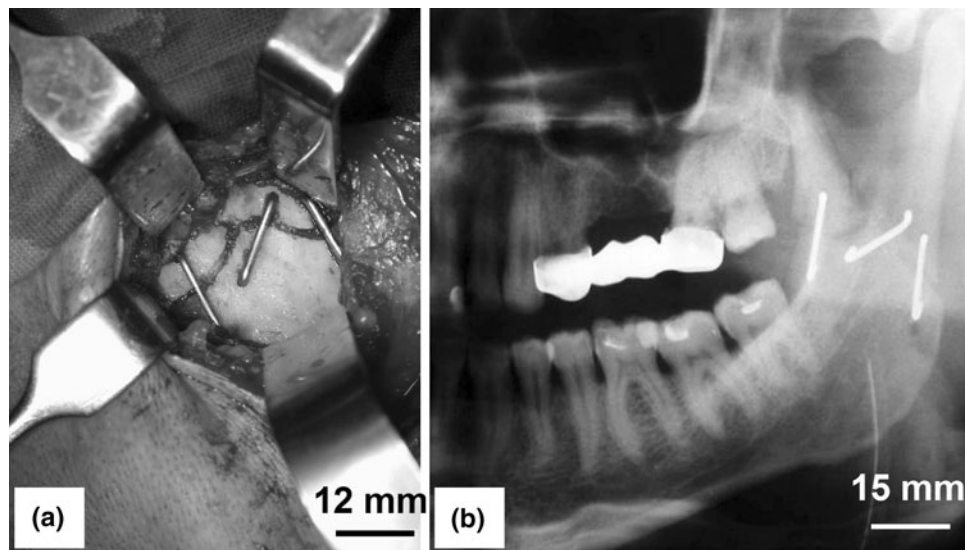


Fig. 16 Joining of condyle mandible fracture by NiTi shape memory staples (a) and radiogram marked after operation (b)

4. Conclusions

NiTi staples which recover the desired shape under the influence of the human body heat can be prepared from the studied wires after cold drawing and annealing in the temperature range from 400 to 500 °C for 15-30 min.

After cold drawing with 40-50% deformation and annealing at 400 °C for 30 min very good superelastic properties of TiNiCo wires were obtained.

Staples from the wire of a small diameter can be used for fixation of bone fractures as superelastic staples which may be mechanically opened at a room temperature with a pincer and inserted into the drilled bone holes. Those staples we proposed for fixation of face bone fractures, for example: for fixation of zygomatic bone fracture or the Le Fort I face bone fracture.

The use of shape memory and superelastic staples instead of titanium plates and screws is easier and shortens the operation time.

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